SPECTROSCOPY SOFT AND TENDER (SST)

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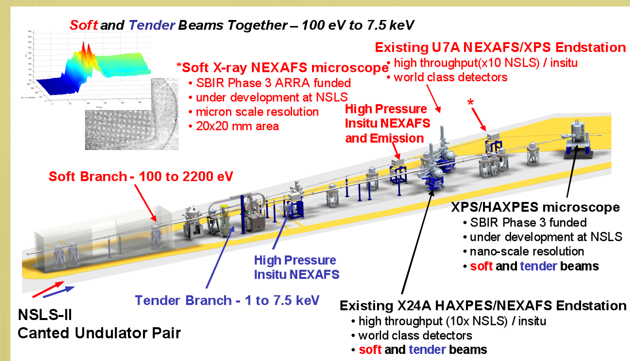
National Institute of Standards and Technology

BROOKHAVEN NATIONAL LABORATORY

NATIONAL SYNCHROTRON LIGHT SOURCE II type-II beamlines • funded by NIST

TECHNIQUES AND CAPABILITIES

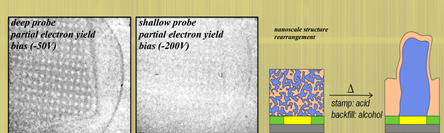
- The mission of SST is to apply X-ray Photoelectron Spectroscopy (XPS) and Near Edge X-ray Absorption Fine Structure (NEXAFS) spectroscopy to the materials science of important societal challenges in energy, health, environment, and national security; to establish structure/function relationships in advanced materials, often at the nanoscale; to accelerate the development of new materials into devices and systems with advanced functionality; and to promote innovation which enhances US industrial competitiveness.



- SST will have 6 unique world class NEXAFS/XPS experimental stations:
  2 full field microscopes, 2 automated high-throughput stations, and 2 in-situ high pressure stations.

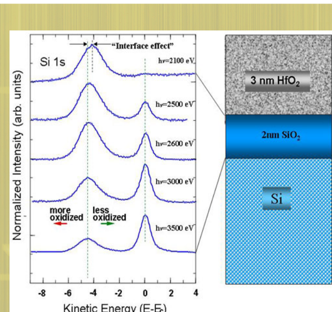
- Soft and tender beams enabling a continuous selection of X-rays from 100 eV to 7.5 keV at a common focal point in a single experiment (unique capability, enhancing depth selectivity in XPS or HAXPES).

APPLICATIONS



Accelerating Organic Photovoltaics with Soft and Tender Spectroscopy and Microscopy

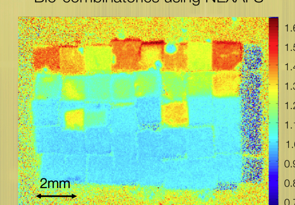
The commercialization of OPV technology has been slowed by two key technical challenges: enhancing the power conversion efficiency, and preventing aging-related performance loss. To address these challenges requires precise measurements of materials composition and molecular orientation with sub-100 nm spatial resolution. These needs will be met by NIST's new soft and tender X-ray microscopes and beamlines for NSLS-II, which promises to greatly accelerate the development of high-efficiency, long-lasting solar modules with the potential to build a new domestic solar cell manufacturing industry in the U.S. and transform our energy production landscape in the coming decades.



Nanoscale Spectroscopy for Next Generation Semiconductor Microelectronics: CMOS and Beyond

Imaging the structure and chemistry of buried layers and interfaces of real device architectures is essential towards ultimate Complementary Metal Oxide Semiconductor (CMOS) scaling and beyond. Above shows Si 1s spectra from 3nm HfO_2/2nm SiO_2 sample recorded with variable kinetic energy high-energy XPS (VKE-XPS) illustrating depth profiling and an interface effect near the HfO_2/SiO_2 interface.

Bio-combinatorics using NEXAFS



Designing Biomaterials with High Throughput Screening: Using the NIST NEXAFS Microscope

Interactions between solid surfaces and the biological environment play an important role in many areas of medicine. Examples include biocompatibility of implants, biomolecule separations, bacterial-induced corrosion, and biosensors. The NEXAFS Microscope allows for unprecedented high throughput screening of samples for biomedical applications. We rapidly evaluated different self-assembled monolayer (SAM) preparation methods by imaging arrays of 45 samples with a surface area of 1 mm² each (above, azobenzene surface density image of 1 hour was equivalent to the data typically collected during five days of serial data acquisition).

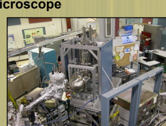
NIST INVESTMENT IN SST *(at NSLS for NSLS-II)*

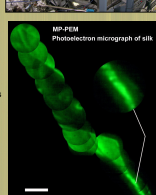
XPS Full Field Magnetic Projection Microscope

Goal: (3D) chemical mapping of the structure of nanomaterials and nanodevices at all points within their volume.

✓SBIR Phase-III collaboration between NIST and R. Browning Consultants, development at NSLS(U4A).

- Super conducting magnetic projection lens
- 3 Patents granted, 4 Patent Applications.
- 100nm spatial resolution.
- Major improvement: Laboratory based Imaging XPS systems throughout the USA and world.(Lab spatial resolution 5000 nm, fixed X-ray energy)
- No high voltages; samples can be rough or insulating, large depth of field.



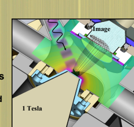


At NSLS-II: Nanoscale spatial and spectral XPS imaging over the full kinetic energy (100 eV to 7.5 keV).

✓ Receive soft and/or tender beams via two KB mirror pairs that have a common focal spot of 13 μm.

✓ Tuning the sensitivity from surface to bulk length scales ideally suited for photovoltaics and microelectronics.

Large Area Imaging NEXAFS Microscope



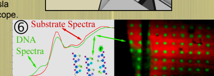
Goal: NEXAFS full field microscope producing highly efficient, spectroscopic chemical and orientation maps of gradient samples, combinatorial arrays.

- **e.g. 1000s of compositional catalyst samples at once, and device arrays up to 4 cm² with micron scale resolution.**

✓SBIR working prototype at NIST's U7A beamline: 1 Tesla magnetic projection partial electron yield full field microscope.

- Image: 13x18 mm at 50 μm spatial resolution.
- Large depth of field (rotatable sample at right).

✓ Science case examples: Microscope images of combinatorial arrays of **ss-DNA** (right), batteries, OPV marine anti fouling designer surfaces and more.

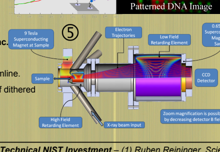


NEXAFS microscope for NSLS-II ARRA funded (FY09 NIST SBIR Phase III, Synchrotron Research, Inc.)

✓ Superconducting magnet (8T)

✓Under construction, commissioning at NSLS U8B beamline.

✓ At NSLS-II, receive soft x-rays (100-2200 eV) a pair of dithered mirrors, 4x4 cm high flux sample illumination.

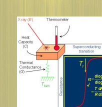


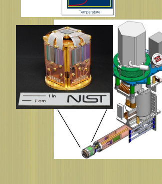
Soft x-ray Emission and NEXAFS : Micro-calorimeter

Micro-calorimeter High-resolution Soft X-ray Detector: Soft x-ray Emission and NEXAFS Spectroscopy at beamline U7A for NSLS I&II

Major Accomplishments:

- NIST-Boulder Quantum Sensors Project collaborating with NIST-Synchrotron Methods Group at NSLS
- Novel soft x-ray-fluorescence spectrometer, no wavelength-dispersive element, based on a array of 256 superconducting transition-edge microcalorimeters.
- **256 pixels, 200 um, and each capable of receiving up to 100 x-ray counts per second with energy resolution of 1 eV (FWHM); the total instrument will have collecting area of 10 mm² and count rate of 25 kHz**
- Fully-populated detector snout, has received first-light photons from a tube source at NIST-Boulder in July, 2010





Key Capabilities:

✓ High-resolution x-ray fluorescence (XRF) spectroscopy, like x-ray photoelectron spectroscopy (XPS), sensitive to occupied electronic states **(measurement of HUMO).** XRF probes deeply buried layers (100nm) from which photoelectrons cannot escape.

✓ Also for reducing background in FY NEXAFS **(measurement of LUMO).** Crucial for studying layer-interface chemistry that determines device properties. (e.g. gatestacks, organic PV, catalysts)

Technical NIST Investment *– (1) Ruben Reininger, Scientific Answers and Solutions - Optical Design, (2) FMB Oxford Initial conceptual CAD Model, (3) Tender DCM and VLSPGM, purchased*